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Fast pyrolysis of Douglas fir by using tilted-slide reactor and characteristics of biocrude-oil fractions

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ABSTRACT

A tilted-slide fast pyrolyzer was designed for the large-scale production of biocrude-oil. Woody biomass (Douglas fir) was fed into the reactor under different experimental conditions, including reaction temperature and biomass feeding rate, in order to study the biocrude-oil yield and its characteristics. One spray type condenser was used to condense the biocrude-oil by direct contact heat transfer and three shell and tube type condensers were used to collect biocrude-oil by indirect contact heat transfer. The highest biocrude-oil yield (61.9 wt.%) was obtained at 490 °C and a feeding rate of 12.5 kg/h, with most biocrude-oil was collected in the spray type condenser. The characteristics of the biocrude-oils obtained from each condenser were scrutinized and compared, and GC/MS analysis was also conducted to ascertain the chemical components of the biocrude-oil thus obtained.

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1. Introduction

Biomass is an important resource for human beings. We utilize biomass materials for a long time to generate heat, to build housing, to manufacture paper, and so on. Although fossil fuels have been predominantly used over the last 100 years, biomass was a major energy source in everyday life before the industrial revolution in the 19th century. However, due to the depletion of fossil fuels and environmental problems, renewable energy derived from biomass is being actively studied nowadays.

Techniques of converting biomass to renewable energy are categorized into biochemical and thermochemical processes [1,2]. For reference, biocrude-oil is a liquid fuel which is produced by a thermochemical conversion process known as fast pyrolysis [3]. Biocrude-oil by fast pyrolysis is considered a promising renewable energy source because its production cost is lower than that of other techniques [4], and it can be directly applied to oil burners, diesel engines, gas turbines and boilers [3]. Hence, many study groups around the world have participated in biocrude-oil production projects [4].

Previous studies on the fast pyrolysis of biomass tended to focus on product yield and biocrude-oil properties [2,5]. For reference, woody biomass has usually been used to understand the fast pyrolysis of biomass [5]. Furthermore, other kinds of biomasses are used to study biocrude-oil quality depending on each biomass [5,6]. However, recent studies have focused on the utilization and quality improvement of biocrude-oil or on reactor scale-up [4]. According to previous studies, biocrude-oil can be applied to conventional combustors although it has some problems such as high viscosity, solid residue, water content and poor combustion behavior [5]. Hence, additional processes aimed at producing biocrude-oil of a higher quality have been adopted, including physical and chemical upgrading techniques [4], torrefaction before fast pyrolysis [7], and biocrude-oil fractionation [8]. Reactor scale-up is essential for the commercialization of biocrude-oil production. Using scale-up, biocrude-oil can be mass-produced and production costs can be significantly reduced. As such, many research efforts aimed at reactor scale-up and cost reduction are being conducted at present [4].

The pyrolysis reactor was originally devised for slow pyrolysis to produce charcoal in ancient times [9]. In order to produce charcoal, a fixed bed reactor was mostly employed [9]. However, contemporary pyrolysis studies have focused on liquid production technology to produce fuel with a higher energy density [3]. For reference, a fixed bed reactor is not suitable for the fast pyrolysis of biomass owing to the long residence time and low heating rate [9,10]. According to a previous study with a fixed bed reactor [10], the biocrude-oil yield was relatively low and its quality was too poor to allow its use as a fuel. Hence, new types of pyrolyzers were devised for biocrude-oil production [4].

A fluidized bed reactor is generally used for fundamental studies of the fast pyrolysis of biomass [3], as it confers certain advantages such

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Table 1
Proximate analysis of Douglas fir.

Water content (wt.%)	Volatile (wt.%)	Fixed carbon (wt.%)	Ash (wt.%)
7.05	80.41	12.26	0.28

Table 2
Elemental analysis of Douglas fir (dry and ash-free basis).

C	H	O[a]	N	S
49.07	6.05	44.78	0.1	0

[a] By difference.

as good solid mixing, a high heating rate and simple design [3]. Moreover, a short residence time can be easily obtained by controlling the fluidization gas flow rate, and it is relatively easy to scale up [3]. There are some of the reasons why many research groups opted for a fluidized bed fast pyrolyzer [3]. However, it has been pointed out that the fluidized bed reactor has various disadvantages including its large size and the elevated construction cost caused by the fluidization gas supply. From previous studies [4,11,12], novel pyrolyzers such as the circulating fluidized bed pyrolyzer, the ultra rapid pyrolyzer, the ablative pyrolyzer, the rotating cone pyrolyzer, the augur pyrolyzer and the vacuum pyrolyzer were introduced for scale-up and biocrude-oil production. With the exception of the circulating fluidized bed pyrolyzer, they all have different operating processes. The tilted-slide reactor in this study also has its own operating process. Biomass and hot sand are slid down through the tilted reactor by gravity. In the reactor, they are mixed together and biomass is simultaneously decomposed during the sliding down process.

Numerical studies of fast pyrolysis and heat transfer in this reactor were carried out in earlier studies [13,14]. Hence, in this paper, fast pyrolysis of woody biomass was conducted using a tilted-slide fast pyrolyzer to study the biocrude-oil yield and quality. In addition, the biocrude-oils thus obtained were scrutinized and compared with typical biocrude-oil data.

2. Experimental

2.1. Raw material

Pulverized woody biomass (Douglas fir) gathered from a woodworking plant in the Daejeon area of South Korea was used in this study. It was sieved using meshes of 1 and 2 mm, and then dried in an oven at 105 °C for 24 h to make its moisture 2–3 wt.%.

Table 1 shows the proximate analysis of the Douglas fir used in this study. The naturally dried woody biomass consisted of 7.05 wt.% water content, 80.41 wt.% volatile, 12.26 wt.% fixed carbon and 0.28 wt.% ash. Table 2 shows the elemental composition of the raw material. The higher heating value (HHV) of the pulverized Douglas fir was 18.88 MJ/kg and its bulk density was 172.8 kg/m^3. Fig. 1 shows the thermogravimetric analysis (TGA) and differential thermogravimetric (DTG) curves of the raw material under nitrogen atmosphere. The heating rate was 5 °C/min. As shown in Fig. 1, the first peak was observed due to water content of biomass [15]. The results of the analysis indicate that the Douglas fir decomposed well at 340 °C. That phenomenon occurred due to decomposition of hemicelluloses and cellulose in Douglas fir [16]. The TGA and DTG curves were similar to the previous studies [15,16].

2.2. Experimental setup

2.2.1. Tilted-slide fast pyrolyzer

Fig. 2 shows the experimental setup of the tilted-slide pyrolyzer used in this study. The reactor was made of SUS 316 and the inner dimensions of the reactor are shown Fig. 3. As shown in Fig. 2, 25 kg of hot sand were put in a sand hopper and then passed into the reactor via a slit. The sand from the hopper flowed inside the tilted reactor like a fluid at a constant rate. The biomass in the biomass hopper was fed into the reactor by two screw feeders at constant feeding rate. The biomass fed into the reactor came into contact with the hot sand at the top of the reactor, and then they were mixed together in the reactor. Therefore, the biomass is instantly decomposed into volatiles, char and non-condensable gases under the O_2 free atmosphere in the reactor. For reference, an induced draft fan was placed at the outlet of the last condenser to lower the pressure in the reactor and make the volatiles flow into the condensers.

The experimental conditions are tabulated in Table 3. Reaction temperatures (440, 490, 540 °C) were chosen to determine the optimum temperature for fast pyrolysis of the Douglas fir. As shown in Fig. 2, the reaction temperatures were measured by a thermocouple at the top point where biomass met hot sand inside the reactor. Another thermocouple was installed at the bottom point to measure the temperature gradient between top and bottom point. The temperature difference between the two point was 50 °C when the biomass and sand flowed from the top of the reactor to the bottom of that. The sand which lost energy by heat transfer to biomass went to a

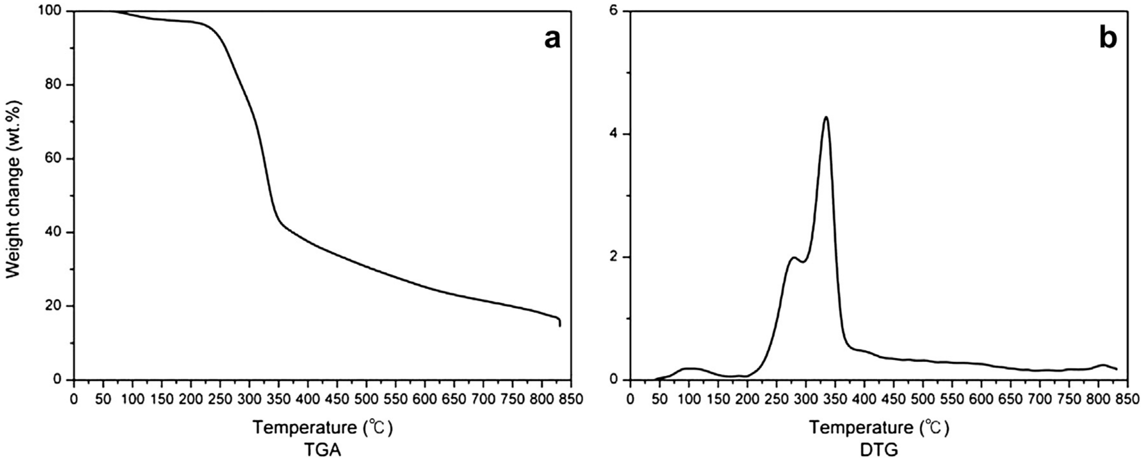

Fig. 1. Thermogravimetric analysis of Douglas fir.

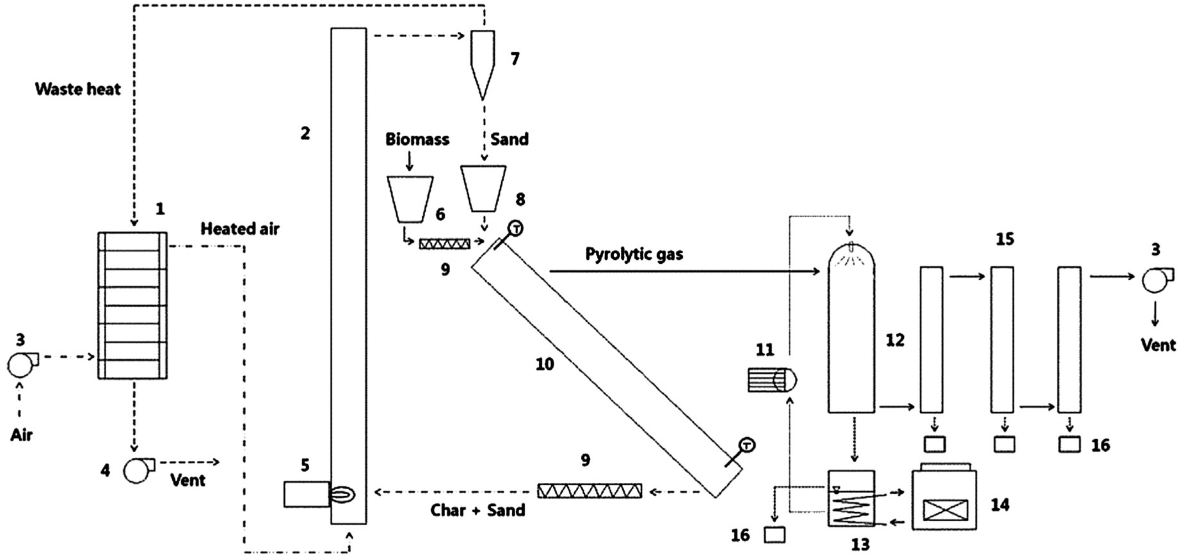

Fig. 2. Schematic diagram of the tilted-slide fast pyrolyzer: (1) Heat exchanger (2) Circulating fluidized bed combustor (3) Forced draft fan (4) Induced draft fan (5) Oil burner (6) Biomass hopper (7) Cyclone (8) Sand hopper (9) Screw feeder (10) tilted-slide reactor (11) Oil pump (12) Spray type condenser (13) Biocrude-oil container (14) water chiller (15) Shell and tube condenser (16) Biocrude-oil bottle.

circulating fluidized bed combustor to gain energy again. A detail explanation about that was in followings. The biomass feeding rate was varied from 10 to 15 kg/h to determine the optimum biomass capacity of the tilted-slide reactor used in this study. The total amount of biomass fed in each experiment was 50 kg.

2.2.2. Biocrude-oil collection

As shown in Fig. 2, the pyrolysis gas initially enters the four condensers for condensing volatiles. One spray type and three shell and tube type condensers were employed to collect the biocrude-oil. As shown in Fig. 2, the spray type condenser rapidly quenched the volatiles at the moment they started coming out of the reactor. As shown in Fig. 2, the biocrude-oil was circulated by an oil pump in the condenser. Therefore, the biocrude-oil was put into a biocrude-oil container located at the bottom of the condenser before conducting each experiment. The biocrude-oil was sprayed at the top of the condenser and condensed volatiles into biocrude-oil. The condensed biocrude-oil was collected in the container by gravity and circulated again to condense biocrude-oil. The produced biocrude-oil flowed out through a hosepipe and was then collected in a bottle. As shown in Fig. 2, a coil heat exchanger pipe was installed in the biocrude-oil container to cool the produced biocrude-oil. The shell and tube type condensers were setup to collect the biocrude-oil which was not condensed by the spray type condenser. During its passage through the condensers, the biocrude-oil was collected in the bottles by gravity.

2.2.3. Char combustion and waste heat recovery

The char produced after the fast pyrolysis of the biomass flowed down to the bottom of the reactor with sand by gravity, and was then fed into a 6 m circulating fluidized bed combustor by a screw feeder. As shown in Fig. 2, the burning char particles transmitted

Table 3
Experimental conditions in this study.

	Case 1	Case 2	Case 3	Case 4	Case 5
Reaction temperature (°C)	440	490	540	490	490
Biomass feeding rate (kg/h)	10	10	10	12	15

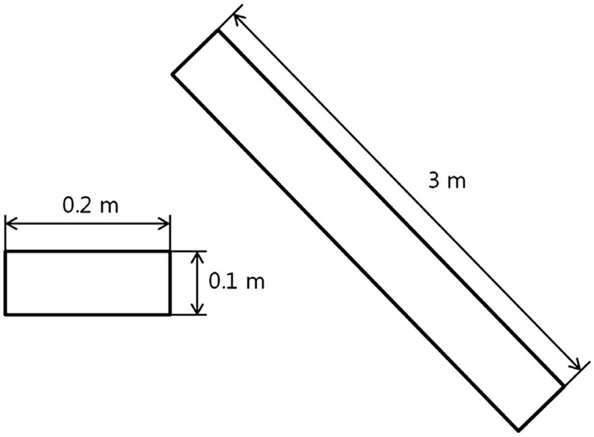

Fig. 3. Inner dimensions of tilted-slide reactor.

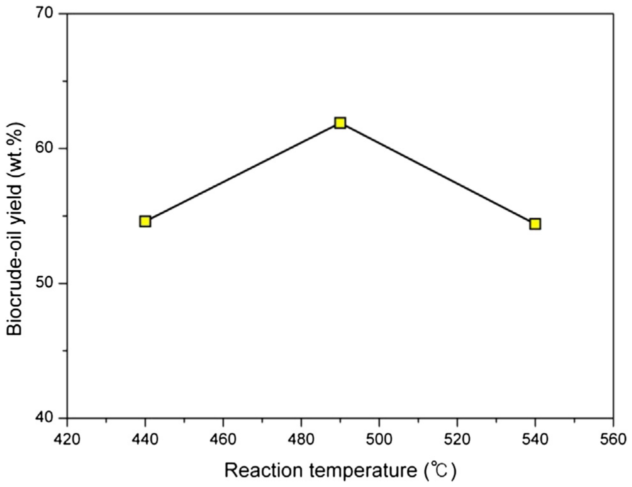

Fig. 4. Biocrude-oil yield with reaction temperature.

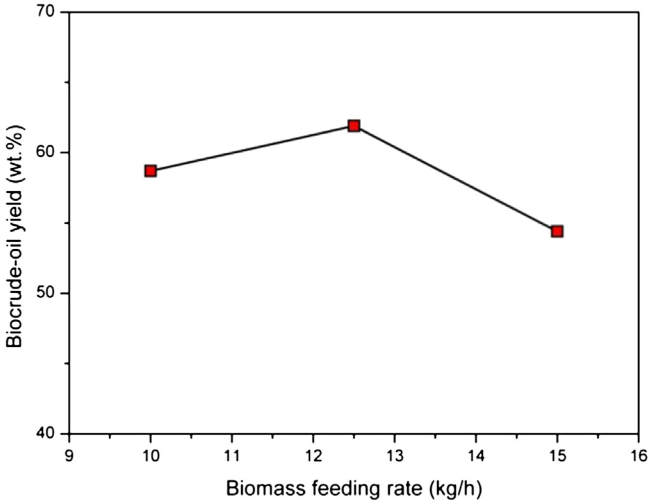

Fig. 5. Biocrude-oil yield with biomass feeding rate.

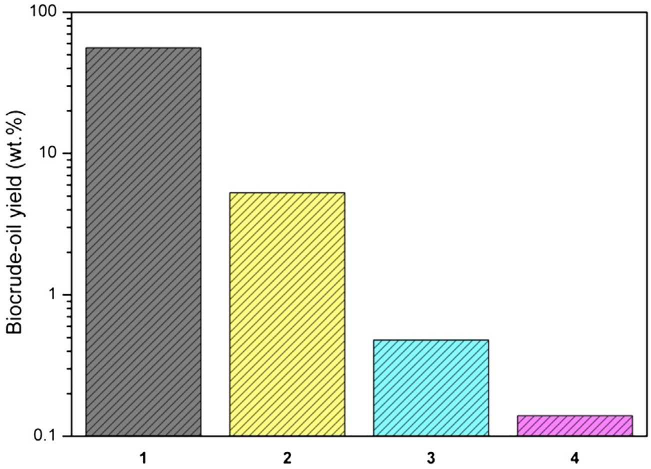

Fig. 6. Biocrude-oil yields from each condenser.

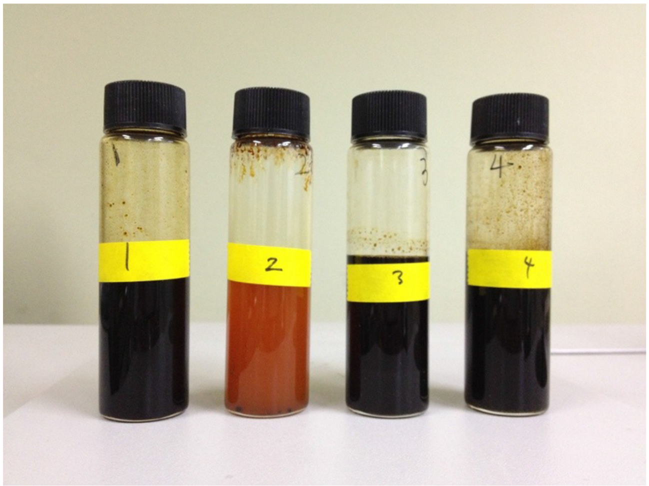

Fig. 7. Sample biocrude-oil collected from each condenser.

Table 4
Characteristics of biocrude-oil.

Condenser no.	HHV (MJ/kg)	Water content (wt.%)	Viscosity (cP at 20 °C)	Solid residue (wt.%)	Ash (wt.%)	Density (kg/L)	pH
1	17.31	25.41	88.1	0.1	0.13	1.2	1.9
2	n/a	74.19	2.97	0.03	0.07	1	2
3	14.07	39.5	7.56	0.04	0.14	1.2	1.9
4	17.38	26.46	33.2	0.02	0.25	1.2	2.1

heat to the sand while rising up to the top of the combustor. The oil burner was used to supply additional heat to the sand. Hence, only hot sand was gathered by the cyclone located at the top of the combustor, and entered the sand hopper again. As shown in Fig. 2, waste heat from the cyclone was recovered by a heat exchanger. The hot air flowing from the heat exchanger entered the bottom of the circulating fluidized bed combustor to reduce oil consumption in the oil burner.

2.3. *Analytical methods*

The water content of the biocrude-oil was measured using the Karl-Fisher titration method according to ASTM D1744. A LECO AC-350 was employed to measure the higher heating value (HHV) of the biocrude-oil following ASTM D2015. A filter (pore size 0.1 μm) was employed to measure the solid residue in the biocrude-oil. A viscometer (Brookfield, Model DVЦ+Pro) was used to measure the viscosity of the biocrude-oil at 20 °C, and a Thermo Orion 410 was used to measure its acidity. The ash content of the biocrude-oil was determined by combusting it in a muffle furnace at 770 °C for a night and then weighting the residue. A Flash EA 1112 series was used in the elemental analysis of the biocrude-oil. An Agilent 7890N gas chromatograph equipped with a DB-1701 (30 m × 0.25 mm × 0.25 μm) was employed in the GC/MS analysis of the biocrude-oil. The injector temperature was 250 °C, and the oven temperature was initially held at 35 °C for 5 min and then increased to 280 °C (heating rate: 6 °C/min) and held at that temperature for 10 min. Helium gas was employed as the GC/MS carrier gas (flow rate: 1 ml/min).

3. Result and discussion

3.1. *Biocrude-oil yield*

3.1.1. *Effect of reaction temperature*

50 kg of Douglas fir was fed into the reactor at different reaction temperatures. Fig. 4 shows the biocrude-oil yield with the reaction temperature. The highest biocrude-oil yield was obtained at 490 °C. The biocrude-oil yield increased between the temperatures of 440 °C and 490 °C and then decreased when the temperature rose above 540 °C. These experimental results were in good agreement with those previously reported in the relevant literature [3]. It was also noted that the reaction temperature is undoubtedly an

Table 5
Elemental compositions of biocrude-oil from each condenser.

Elements (wt.%)	Condenser no.			
	1	2	3	4
C	44.15	11.62	28.29	42.13
H	7.41	8.49	8.66	7.44
O	47.9	79.61	62.32	49.97
N	0.54	0.28	0.73	0.46
S	0	0	0	0

Table 6
Detected chemical compounds in biocrude-oils.

No.	Chemical components	Area %			
		1	2	3	4
1	Acetaldehyde, hydroxy-	2.4451	18.127	4.0936	
2	3-Buten-2-one, 3-methyl-	0.21491		1.1119	1.7509
3	2-Butanone, 3-methyl-			0.38403	
4	2-Butenal	1.0862		3.5169	
5	Acetic acid	23.071			
6	Hydrazine, ethyl-		23.668		
7	Urea			21.367	
8	2-Furanol, tetrahydro-				0.28481
9	Benzene, methyl-				3.3489
10	2-Propanone, 1-hydroxy-	5.9928	5.2344	4.1661	7.4008
11	3-Penten-2-one, (E)-		0.66227	2.7292	
12	2,3-Pentanedione			1.3675	
13	Toluene			3.0228	
14	Ethylidene acetone; methylpropenyl ketone				4.6703
15	Acetic acid, methyl ester	17.056		0.62722	
16	Propanoic acid	1.2458	1.3845		
17	Propanoic acid, 2-oxo-, methyl ester	0.12952	3.2235	0.41055	
18	2-Cyclopenten-1-one	1.4949	1.5413	2.0496	2.5245
19	Oxirane, methyl-, (S)-	3.7778			
20	1,2-Ethanediol, monoacetate		11.25		1.9946
21	Ethylbenzene			1.4347	2.4206
22	2-Furancarboxaldehyde	3.5038	4.4626	5.5845	3.245
23	p-Xylene			0.80134	
24	Benzene, 1,2-dimethyl-				1.4541
25	2-Propanone, 1-(acetyloxy)-	0.25746		0.33208	0.95773
26	Butyric acid	1.1675			
27	1,2-Cyclopentanedione				4.6127
28	2-Cyclopenten-1-one, 3-methyl-		0.72159	1.276	3.0451
29	2(3H)-Furanone, dihydro-				3.4395
30	Propanal, 2,3-dihydroxy-		3.0867		
31	a,a-Crotonolactone			1.1332	3.0523
32	2-Butanone	0.54776	0.33027		
33	2,5-Furandione, 3-methyl-		0.099443		
34	Acetic acid ethenyl ester		0.27704		
35	2-Furancarboxaldehyde, -5-methyl			0.90187	2.4669
36	3-Methyl-2,5-furandione	1.8255		1.1533	
37	2-Cyclopenten-1-one, 2-hydroxy-			1.3191	
38	2-Cyclopenten-1-one, 3-methyl-			0.72809	2.2753
39	4H-1,3-oxazin-2-amlne, 5,6-dihydro-n-methyl-				2.3155
40	2(5H)-Furanone, 3-methyl-				1.2429
41	2-Cyclopenten-1-one, 2-hydroxy-3-methyl-				0.94331
42	Butyrolactone			1.8125	
43	1,2-Cyclopentanedione, 3-methyl-			0.012014	
44	Phenol		0.84856	1.9161	3.4543
45	Guaiacol		0.40399	0.82878	2.4172
46	Phenol. 2-methyl-		0.40462	1.1664	3.3485
47	2,5-Furandicarboxaldehyde				
48	2(5H)-Furanone, 4-methyl-				2.0415
49	Pentanal	3.2657	1.7653		
50	Guaiacol, 4-methyl-				1.6028
51	Phenol, 2,4-dimethyl-	0.71905	0.51208	1.3716	3.7259
52	Butanal, 3-methyl-			0.018584	
53	4-Methyl-2-oxopentanenitrile	0.7529	0.22779		
54	2-Furancarboxaldehyde, 5-(hydroxymethyl)-	1.0024	0.43976	2.4536	3.3085
55	2,6-Dlmethyl-2,6-octadlen-1-ol			0.19337	
56	Phenol, 2-ethyl-6-methyl-			0.90852	
57	1,4:3,6-Dianhydro-a-d-glucopyranose			1.0746	
58	2-Methoxy-4-vinylphenol			0.34443	
59	Guaiacol, 4-allyl-			0.20598	
60	1,2-Benzenediol	5.234	1.8631	5.2906	4.6201
61	(s)-(+)-2,3-Dideoxyribonolactone			2.0954	
62	Guaiacol, 4-propenyl-cis			1.0741	2.2331
63	1,2-Benzenediol, 4-methyl-	1.9335	0.74955	4.1475	6.4968
64	Vanillin			1.0781	6.4968
65	4-Ethylcatechol			3.2657	
66	D-allose	3.8504	3.9998		
67	3,4-Altrosan	8.2374	8.6194	10.674	
68	Levoglucosan	12.734	4.9301	0.001044	11.263
69	2-Propenal, 3-(4-hydroxy-3-methoxyphenyl)-			0.55628	

important parameter for biocrude-oil yield [3,17]. In cases 2 and 3, the experiments were well conducted. However, in case 1, the char did not burn well in the circulating fluidized bed combustor because more char was produced at the lower temperature [3] and the relatively low-temperature char was fed into the combustor. Therefore, the unburned char entered the reactor again, disturbing the heat transfer from hot sand to biomass. Moreover, the ash content of the char caused the catalytic cracking of volatiles [4], which was another reason for the decrease in the yield of biocrude-oil in case 1.

3.1.2. *Effect of biomass feeding rate*

In this study, the Douglas fir was fed at different feeding rates. Fig. 5 shows the biocrude-oil yield with the biomass feeding rate. The highest biocrude-oil yield was 61.9 wt.% when 12.5 kg/h of biomass was fed into the reactor. According to a previous study, the biocrude-oil yield increases at a higher biomass feeding rate [18]. These experimental results showed a clear agreement with that finding. The experiment in case 4 also performed well. However, in case 5, the unburned char entered the sand hopper with the sand. According to the increase in the feeding rate, more char was produced, and it could not burn well in the circulating fluidized bed reactor causing a similar phenomenon to that observed in case 1. This is the reason why the biocrude-oil yield decreased rapidly at a biomass feeding rate of 15 kg/h.

3.1.3. *Yields of biocrude-oil fractions*

Fig. 6 shows the biocrude-oil yields from each condenser using a logistic scale. From the experimental results, the most biocrude-oil was obtained by the first condenser. As shown in Fig. 6, 56 wt.% of biocrude-oil was collected by the spray type condenser, while 5.28 wt.%, 0.48 wt.% and 0.14 wt.% of biocrude-oil was collected by shell and tube type condensers.

3.2. *Characteristics of biocrude-oil*

The biocrude-oils produced in this study are shown in Fig. 7. The biocrude-oils consisted of a dark brown liquid, except for the biocrude-oil obtained from the second condenser. As shown in Fig. 7, the color of the biocrude-oil obtained from the second condenser was light brown as it contained lots of moisture. For reference, some dark brown lumps which were hardly dissolved in the light brown liquid were observed. In addition, the biocrude-oils produced in this study emitted a smoky odor. Table 4 shows the physical and chemical properties of the biocrude-oils produced in this study. The HHV of the biocrude-oil from the first condenser was 17.31 MJ/kg and its water content was 25.41 wt.%. The HHV of the biocrude-oil was lower than that of the raw material. However, its energy density increased greatly from 3.26 GJ/m^3 (determined on the basis of the bulk density of the raw material) to 20.77 GJ/m^3. These properties are similar to the typical properties of biocrude-oil reported in the literature [3]. The water content of the biocrude-oil collected by the second condenser was 74.19 wt.%. Due to its high water content, its HHV could not be determined and its viscosity was much lower than that of other biocrude-oils. For reference, it would be hard to use it as a fuel for conventional combustors as a fuel due to its high water content. The HHV of the biocrude-oil from the third condenser was 14.07 MJ/kg and its water content was 39.5 wt.%. Phase separation was observed in the biocrude-oil obtained from the third condenser. Heavy biocrude-oil was deposited at the bottom: this phenomenon occurred due to its high water content, and was in good agreement with the findings reported in the literature [19]. It was of lower quality than the widely known typical biocrude-oil [3]. The properties of the biocrude-oil from the fourth condenser were similar to those of the biocrude-oil from the first condenser. The solid residue, ash and density of the biocrude-oil were in good agreement with the properties of typical biocrude-oil [3]. However, the pH of the biocrude-oils was relatively lower than that of typical biocrude-oil [3].

Table 5 shows the elemental analysis of the biocrude-oils produced in this study. As is well known in the literature [3], biocrude-oils produced from Douglas fir are highly oxygenated. In particular, the oxygen content of the biocrude-oil from the second condenser was 79.61 wt.%. Also, the carbon content of the biocrude-oil was in the range of 11.62–44.15 wt.%. It was closely related to the HHV of biocrude-oil. Sulfur was not present in the biocrude-oils.

Table 6 shows the GC/MS analysis of the biocrude-oils: some 69 chemical components were detected in the biocrude-oil. As shown in Table 6, the biocrude-oils produced in this study were rich in levoglucosan which was derived from cellulose [6]. The biocrude-oils were also found to contain phenols derived from lignin [6].

4. Conclusions

The fast pyrolysis of Douglas fir was carried out using a tilted-slide reactor for biocrude-oil production. The fast pyrolysis of Douglas fir proceeded smoothly in the reactor, and biocrude-oil was steadily produced during each experiment. The highest biocrude-oil yield was 61.9 wt.% at 490 °C, with a biomass feeding rate of 12.5 kg/h. The char did not burn well when the reaction temperature was below 440 °C or when the biomass feeding rate was above 15 kg/h. These phenomena caused the decrease of the biocrude-oil yield. The largest amount of biocrude-oil was collected by the first spray type condenser, with the rest collected by the other condensers. The biocrude-oils displayed different properties depending on which condenser they were collected from. In particular, the HHV, water content and viscosity of the biocrude-oil varied significantly depending on the condenser. Judging by the results, the properties of the biocrude-oil collected from the first and fourth condensers agree with the typical properties of biocrude-oil obtained from woody biomass. Hence, it could be directly applied to conventional combustors. Conversely, the biocrude-oil obtained from the second and third condensers was rich in moisture, making it less suitable as a fuel than other biocrude-oils.

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